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**NANOPARTICLE-WETTED RELAYS:
RECONFIGURABLE SURFACES FOR
ENERGY TRANSMISSION CONTACTS
(PREPRINT)**



**A.A. Voevodin, R. Vaia, S.T. Patton, S. Diamanti, M. Pender,
M. Yoonessi, J. Brubaker, J.J. Hu, J.H. Sanders, B.S. Phillips,
and R.I. MacCuspie**

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AIR FORCE MATERIEL COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OH 45433-7750**

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*//Signature//

ANDREY A. VOEVODIN, Project Engineer
Nonstructural Materials Branch
Nonmetallic Materials Division

//Signature//

STEPHEN L. SZARUGA, Acting Chief
Nonstructural Materials Branch
Nonmetallic Materials Division

//Signature//

SHASHI K. SHARMA, Acting Deputy Chief
Nonmetallic Materials Division
Materials and Manufacturing Directorate

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14. ABSTRACT Performance and reliability of dynamic physical contacts between two solid surfaces has challenged technologists from pre-Hellenistic pulleys and Da Vinci mechanisms for transmission of mechanical energy to modern switches and relays for transmission of electrical energy, currently a \$4B global industry that impacts telecom and mobile phones, automotive, aerospace and consumer products. Local oscillations in the stress, temperature and electrostatic potential during a contact cycle result in the evolution of the topology, chemistry and physical properties of the two surfaces, degrading the characteristics of the junction with service. These challenges are especially acute in ultra-fast (MHz) micro electromechanical system (MEMS) relays requiring high current ($>10 \mu\text{A}$), low impedance ($<2 \Omega$) operation over billions of cycles. Various surface modification approaches, such as gold alloys and refractory coatings and self-assembled monolayers, have been unsuccessful in enabling the maintenance of these multifunctional (low adhesion, low resistivity) contacts. <i>(Continued on back of page)</i>					
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14. ABSTRACT (concluded)

Taking inspiration from 1940s mercury-wetted electrical contact relays and current nanoparticle organic solutions, here we demonstrate that noble metal nanoparticle liquids (NPLs) provide reconfigurable and replenishable surface asperities that extend the durability by 10 to 100 times (10 μ A to 1 mA, respectively) without the inherent toxicity of mercury or capillarity that limits relay miniaturization. These non-volatile NPLs are made of 5-20 nm Au and Pt nanoparticles with organic coronas consisting of surface tethered ionic liquids. The nanoscopic size and corona fluidity are critical in providing sufficient electrical conductivity through nanoparticle jamming while maintaining a low contact adhesion by dynamically restoring a nanoscopic asperity texture via liquid surface migration. The general extension of nanoparticle-wetted reconfigurable surfaces for impact, sliding, or rolling will help to realize self-maintained dynamic physical interfaces with increased reliability and reduced losses for transmittance of electrical and mechanical energy.

Nanoparticle-Wetted Relays: Reconfigurable Surfaces for Energy Transmission Contacts

A. A. Voevodin¹, R. Vaia¹, S. T. Patton¹, S. Diamanti¹, M. Pender^{1,2}, M. Yoonessi^{1,2}, J. Brubaker¹, J. J. Hu¹, J. H. Sanders¹, B. S. Phillips¹ & R.I. MacCuspie¹

¹*Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright-Patterson Air Force Base, Ohio 45433, USA*

Performance and reliability of dynamic physical contacts between two solid surfaces has challenged technologists from pre-Hellenistic pulleys and Da Vinci mechanisms for transmission of mechanical energy¹ to modern switches and relays for transmission of electrical energy, currently a \$4B global industry that impacts telecom and mobile phones, automotive, aerospace and consumer products². Local oscillations in the stress, temperature and electrostatic potential during a contact cycle result in the evolution of the topology, chemistry and physical properties of the two surfaces, degrading the characteristics of the junction with service. These challenges are especially acute in next generation ultra-fast (MHz) micro electromechanical system (MEMS) relays requiring high current ($>10\ \mu\text{A}$), low impedance ($<2\ \Omega$) operation over billions of cycles³⁻⁵. Various surface modification approaches, such as gold alloys and refractory coatings^{6,7} and self-assembled monolayers^{8,9}, have been unsuccessful in enabling the maintenance of these multifunctional (low adhesion, low resistivity) contacts. Taking inspiration from 1940's mercury-wetted electrical contact relays¹⁰ and current nanoparticle organic solutions¹¹, here we demonstrate that noble metal nanoparticle liquids (NPLs) provide *reconfigurable and replenishable* surface asperities that extend the durability by 10 to 100 times ($10\ \mu\text{A}$ to 1 mA, respectively) without the inherent

²Present address: Michelin Americas Research and Development Corporation, 515 Michelin Road, Greenville, South Carolina 29605, USA.

toxicity of mercury or capillarity that limits relay miniaturization. These non-volatile NPLs are made of 5-20 nm Au and Pt nanoparticles with organic coronas consisting of surface tethered ionic liquids. The nanoscopic size and corona fluidity are critical in providing sufficient electrical conductivity through nanoparticle jamming while maintaining a low contact adhesion by dynamically restoring a nanoscopic asperity texture via liquid surface migration. The general extension of nanoparticle-wetted reconfigurable surfaces for impact, sliding, or rolling will help to realize self-maintained dynamic physical interfaces with increased reliability and reduced losses for transmittance of electrical and mechanical energy.

Figure 1 summarizes the concept of nanoparticle-wetted relays based on a reconfigurable nanoparticle contact. Two conductive gold surfaces are coated with a monolayer of gold NPL, thus minimizing any chemical or electrochemical reactions. The NPL complements the inherent surface roughness, generating additional electrically conductive asperities. As the two surfaces approach, particle-particle as well as particle-surface contacts form before surface-surface contact. The higher modulus of the nanoparticles relative to the intervening space arrest further approach of the two surfaces. Current will preferentially flow through these conductive bridges. This in essence, mimics surface reconfiguration of mercury wetted contacts, which since the 1940's was a "gold standard" for dynamic high speed current switches¹⁰ but is currently on the verge of extinction due to mercury toxicity¹². Unlike mercury wetted contacts, the particle size and density in NPL can be optimized with regard to the inherent contact surface roughness such that the nanoparticle created mean-surface roughness balances Joule heating (typical failure for small effective contact areas of rough surfaces) and van der Waals adhesion (typical failure for large effective contact areas of smooth surfaces). Upon retraction of the surfaces, the conductive particle bridges will break locally, regenerating the original nanoparticle coated electrode. The presence of a soft corona

around the nanoparticles prevents particle agglomeration. Finally, NPL fluidity in the contact plane i) facilitates local rearrangements of the particles to maximize the conductive contact area, ii) provides for dynamic replenishment from external reservoirs, and iii) carries away excess heat from the contact zone.

The key component of these nano-enabled reconfigurable conductive contacts is the NPL, a monolithic hybrid material comprised of an inorganic nanosized metallic core and an organic, low-viscosity corona. Maximizing the molecular dynamics within the corona minimizes the volume of corona necessary to impart macroscopic fluidity to the nanoparticle assembly. These characteristics have been previously reported at elevated temperature for alkane thiol passivated gold nanoparticles^{13,14} and more recently by Giannelis and co-workers^{15,16}. NPLs with 20 nm gold particles and 5-to-10 nm platinum particles (Figure 2) discussed here and described in the Methods section are based on an inverted electrostatic scheme from these later studies^{15,16}. These NPLs exhibit a zero volatile organic content, similar to ionic liquids, due to the electrostatic character of the organic component.

The potential of the NPLs in eliminating the two most typical micro-electrical contact failure mechanisms^{3,4} by adhesion at low (10 μ A) and melting at high (1 mA) current switching regimes was examined using a test apparatus developed to imitate loads, displacements, currents, and frequencies of RF MEMS switches with *in-situ* monitoring of contact adhesion forces and electrical resistively fluctuations^{4,17}. The experiments were performed under a dry nitrogen environment at load and current conditions described in the Methods section and selected to mimic that used in packaged MEMS devices. The two targeted degradation mechanisms severely limit the service life of the Si or GaAs based MEMS switches, which use traditional gold plated contacts^{3,4}.

Figure 3 demonstrates that the use of an NPL extends the durability of the switch contact by at least 10-100 times relative to the current technologies (bare gold-plated contacts or molecular level passivation). Up to 10^6 cycles, the contact resistance (Ω) of the relay with the gold NPL is below the failure cut-off level of $2\ \Omega$ for both current regimes. Platinum NPLs show a similar performance. For contact resistance above $2\ \Omega$, high frequency operation is compromised and too much energy is dissipated in the current transfer³.

First consider the extreme conditions of high current switching. Traditional gold-plated contacts fail around 10^3 cycles due to excess local heating which melts regions within the contact area³. Upon retraction, these molten regions are drawn, forming bridges between the contacts and shortening the open position of the switch. The lower left image of Figure 3 shows evidence of extensive surface melting and topography degradation, as well as pronounced deviation from a square wave contact resistance switch cycle. In contrast, only minor changes of the contact surface are observed when the Au NPL was applied (lower right image of Figure 3). The absence of surface degradation correlates with the retention of a square shaped off-on-off contact switch cycle, where the resistance in the closed position is less than $1\ \Omega$. Irreversible degradation of the surface begins between 10^3 and 10^4 cycles and the contact resistance nears the failure criterion by 10^5 cycles. However, the ideal square contact resistance per period is maintained up to 10^5 cycles - 100 times better than that for the traditional bare gold-plated surfaces.

In the lower current switch regime of $10\ \mu\text{A}$, contact heating is insignificant, however surface asperities are mechanically flattened during multiple cycling³. This increases the real contact area, multiplying van der Waals surface adhesion forces, and leading to failure in the closed position, referred to as a contact stiction. For bare gold-plated surfaces, the adhesion forces were about $1\ \mu\text{N}$ at the beginning of the experiment and

gradually increased by 3 orders of magnitude to 1 mN at 10^5 cycles. The flattening of the surface asperities also promoted chemical interactions, which are believed to be responsible for the deviation of contact resistance cycle from a square shaped wave³. In contrast, the adhesion force for the NPL coated contacts fluctuated between 100-500 μ N over the entire 10^6 cycles, at which time the experiments were stopped due to time constraints. The ideal square contact resistance period was maintained for the entire duration of the tests.

A recent alternative approach with a self-assembled monolayer (SAM) organic coating⁹, proved unsatisfactory due to hydrocarbon decomposition or removal of SAM layers. In contrast, NPL performance was not limited by a degradation process, even though the corona of the nanoparticle liquids are comprised of saturated hydrocarbons. At lower current, initial cycling of SAM coated electrodes improves the contact resistance, probably due to partial removal of the hydrocarbons. Ultimately though, the life-to-failure is comparable to that of the uncoated analog and substantially less than with NPLs.

The hypothesized mechanism for enhanced relay performance with the NPLs in Figure 1 is consistent with post-mortem examination of the contact area after 10^5 cycles at the high current regime. In contrast to the extensive melting and interconnecting nano-wire formation of bare contacts, only 50-200 nm molten spots could be observed in the contact area with NPLs (Figure 4, lower image). The molten areas are restricted to 5-to-10 times the size of the initial nanoparticles and comparable to the size of the particle clusters (Figure 2). This observation is consistent with nanoparticles jamming between gold electrodes and serving as a path for the current flow, which leads to localized nanoparticle melting. The ionic and non-volatile liquid surrounding the nanoparticles is thought to restrict the melting to the volume of individual particles and clusters. This molten volume is insufficient to form interconnecting nano-wires across the 1-2 μ m

contact separation. Also consistent with the formation of smaller, localized hot spots, is the reduced impact of hydrocarbon degradation as noted by the previously discussed comparison of NPL performance to that of SAMs. Nanoparticles that are not molten or substantially deformed are still present, implying that sufficient contact asperity is present for continual mediation of the adhesion forces.

Furthermore, lower magnification imaging and Auger analysis of the contact and surrounding area demonstrated NPL migration toward the contact zone and particle depletion from the surrounding areas (Figure 4). Using Pt NPLs to provide elemental contrast with the Au electrode surface, spatially resolved Auger analysis indicated enhanced Pt content in the contact region and reduced Pt content in the surrounding areas, both relative to the initial uniform Pt distribution. Such redistribution of the nanoparticles during multiple cycling provides a mechanism to replenish particles distorted by melting in the contact area and self-restore contact asperities. This surface self-restoration can be further controlled via adjustment of the corona chemistry and fluidity, as well as optimizing nanoparticle sizes and shapes¹¹ to promote migration.

In summary, noble metal NPLs consisting of 5-20 nm Au or Pt nanoparticles with ionic molecular liquid coronas provided *reconfigurable and replenishable* surfaces with controlled asperities and metallic conductivity that extended the durability, relative to alternative approaches, of relays by 10 to 100 times (10 μ A and 1 mA regimes, respectively). The nanoscopic size, metallic conductivity, and corona fluidity are critical in: i) prevention of adhesion failure by providing polydisperse surface asperities; ii) maintenance of electrical current flow through sandwiched nanoparticles with minimum resistance; iii) restriction of nanowire formation and shortening failure by localization of surface melting; and iv) replenishment of these beneficial characteristics by NPL migration on the surface. These observations resonate with current discussions of friction reduction in sliding contacts with nanoparticles dispersed in organic

solutions¹¹. A recent review of the impact of nano scale asperities in mechanical contacts by Israelachvili et al.¹⁸ indicated that the contact roughness could be the most important factor controlling both surface adhesion forces and contact fracture resistance or endurance. From modelling studies of the roughness in electro-mechanical metallic contacts¹⁹, the effect of the surface asperities is even further exaggerated since they control the path of the current transfer in addition to the surface mechanical and chemical interactions.

Further tailoring of the electrical conductivity, thermal transport, and surface reconfigurability of the NPLs with zero volatile organic content will provide options for a wide range of micro and macro electrical contacts, ranging from low current signal switches to high current relays in alternative energy sources, e.g. ambient based hydrogen fuel cells and space based solar elements. An immediate impact of the NPLs is to unlock the bottleneck of MEMS switch reliability. These relays create a basis for high performance, broad band, long range telecommunication and phased array radar navigation capabilities. In addition to electrical relays, performance of mechanical energy transporting contacts is also dictated by the contact asperity roughness, where the initially optimized state degrades in multiple contact engagements, leading to energy losses and failures. The ability of surface confined NPLs to dynamically reconfigure and replenish surface topography, restore initial asperity roughness and physical-chemical-mechanical properties provides a unique self-maintenance for all energy transmitting interfaces.

METHODS

Preparation of nanoparticle-wetted surfaces. Gold NPLs with a 20 nm diameter were synthesized following the Turkevich Method²⁰ and passivated with an excess (5 fold) of the sodium salt of mercaptoethanesulfonate (MES). The subsequent ruby-coloured aqueous solution was combined with a 2-fold equivalence of a quaternary alkyl

ammonium chloride (Adogen 464). The gold nanoparticles were collected and purified from the resultant two-phase, blue-coloured mixture by repetitive (5-times) centrifugation and re-suspension in water and ethanol. The product was insoluble in water but formed ruby-coloured solutions in toluene as anticipated for dissolution of individual gold nanoparticles of this size. Total gold content varied from 10 to 80 wt% depending on the extent of purification, where the excess mass is attributed to the Adogen surfactant. Small angle neutron scattering and transmission electron microscopy indicated the organic ionic corona was $\sim 1.5\text{-}2$ nm. In a similar fashion, platinum NPLs were produced via the reduction of H_2PtCl_6 in the presence of a 3-fold equivalence of MES with the drop wise addition of a chilled solution of NaBH_4 (170 mM). The ligand exchange step for Adogen was performed identically as above to yield platinum NPLs of $\sim 5\text{-}10$ nm in diameter. NPLs were applied to the gold plated GaAs wafer surfaces by spin coating from a toluene solution.

MEMS contact switch experiments. The switch experiments were performed with ball versus flat contact geometry in a dry nitrogen environment with relative humidity of less than 1%, at peak contact loads of 100-200 μN , and square-wave switching at 5 Hz. This is slower than in real RF MEMS devices, providing accelerated contact testing by an increased total energy transmission per single cycle. The base switch contact surfaces were a gold-plated polished GaAs wafers with a local surface roughness of about 1 nm and a long range undulation of ~ 15 nm over 2 μm period. The counter electrode was a gold-plated steel ball with a diameter of 1.6 mm, providing an apparent initial contact area of $\sim 5 \mu\text{m}^2$. Contacts were engaged in hot switching in two regimes: (1) low current of 10 μA (typical for contact stiction failure); and (2) high current of 1 mA (typical for contact melting/shortening failure). The contact chemistry, size, initial roughness, contact forces, and mode of operation were identical to a typical MEMS switch device. Test apparatus permitted *in-situ* monitoring of contact processes as described previously^{4,17}.

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Author Information The authors declare no competing financial interests. Correspondence and requests for materials should be addressed to A.A.V. (andrey.voevodin@wpafb.af.mil).

Figure 1. Conceptual mechanism for nanoparticle-wetted relays with reconfigurable contact surfaces. For one period (open-closed-open cycle), two gold (Au) coated contacts are brought together and retracted creating an ideal, square contact resistance (Ω) trace as a function of time (s). NPL forms asperities on the contact surfaces in the initial open position. Upon closing, nanoparticles jam, forming metallic bridges for current flow and preventing adhesive interaction of the electrode surfaces. Due to the weak physical attraction between the nanoparticles in NPL, these bridges easily break during separation of the surfaces. Statistically, the deformed and not-deformed particles will be equally distributed on the two surfaces. The in-plane fluidity of

NPL restores contact asperities by particle migration. Note that the particle and contract separation are not drawn to scale.

Figure 2. Scanning electron microscope image of a gold electrode with a spin-coated Au NPL and a schematic of an individual particle.

NPL provides ~10% surface area coverage with individual particles and their clusters. The NPL consists of 20 nm diameter Au cores passivated with an ionic corona (1.5-2 nm) comprised of mercaptoalkyl sulfonate (red) that is charged balanced by a quaternary alkyl ammonium cation (burgundy). The nanoparticles create surface asperities that are larger than the initial roughness of the gold coated contact (~1 nm). Excess ionic organic component of the NPL coats the surface between the nanoparticle islands. Ellipsometry indicated an average coating thickness of ~3-4 nm.

Figure 3. Performance comparison of NPLs versus traditional gold plated and alternative SAM coated contacts.

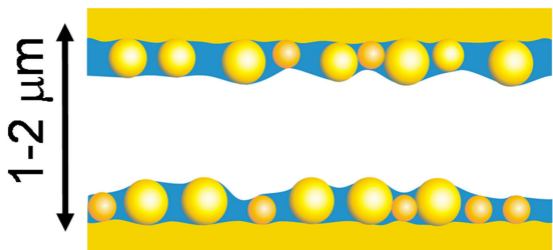
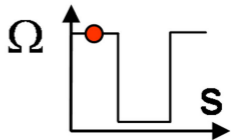
Upper graph compares evolution of the contact resistance as a function of cycles for low (10 μ A, circles) and high (1 mA, triangles) current contact switching. Au NPLs extends the performance 10 to 100 times beyond alternative technologies. Comparable behaviour is observed for Pt NPLs (not shown). Lower images compare the contact region after 10^3 cycles at 1 mA for (1) the failed native gold surface and (2) the robust NPL contact. The insert summarizes the contact resistance change for a single open-close-open cycle. Note the noise-free switching and a lower closed contact resistance in the NPL system (2).

Figure 4. Evidence for suggested mechanisms of NPLs behaviour on reconfigurable surface.

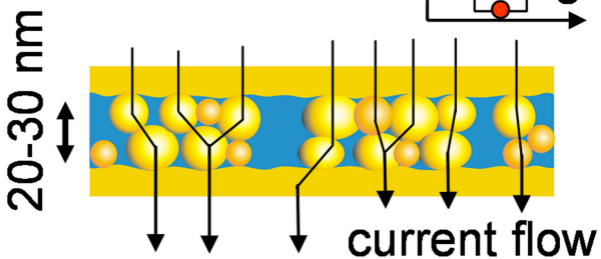
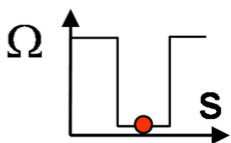
Results provide morphology and chemical analysis of a gold-plated contact area with NPL after 10^5 cycles at 1 mA. Au and Pt NPLs were comparable. High magnification scanning electron micrograph (lower left) reveals localized particle melting and termination of nanowire formation, as well as the presence of un-melted particles. Lower magnification image (top left) of the contact spot shows build up of mass and apparent depletions from the surrounding area. Using Pt NPL to provide elemental contrast, spatially resolved Auger spectra (right) are compared for locations (1) and (2) denoted in

surface morphology micrograph, which confirm Pt particle depletion from the surround area and thus NPL migration to the contact area.

open



closed



open

